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ANALYTICAL METHODS

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TD/5/T43

Findings:A: Solid Phase Extraction

1) The consistency of batches from five different lots of J.T.Baker C-18 SPE columns (100 mg) was examined first. The recoveries of the tri-, tetra- and pentachlorophenols and of dicamba, 2,4-dichlorophenoxypropionic acid, 2,4-D, 2,4,5-T, silvex, 2,4-DB and picloram were determined. Values for the blanks were also determined. The variation between lots was found to be comparable to the variations within lots.

2) Larger SPE columns, 500 mg and 1 g, gave much faster flow rates and allowed more sample volume to be passed before becoming blocked. However, these larger columns gave extremely high levels of interferences detectable by GC/ECD necessitating treatment of the columns prior to use.

3) The volumes of methanol and water used to condition the columns was not critical nor were sample flow rates at practical levels.

4) Very fast elution flow rates were found to reduced the recoveries and also decrease the reproducibility.

5) The effect both of introducing various wash solvents (before and after extraction of the sample) and of varying the eluting solvent composition on the level of interferences is being investigated.

6) The levels of interferences obtained using SPE for PAHs and the influence of the solid phase and eluting solvent on their recoveries was studied.

B: Derivatization of chlorophenols and phenoxy-acids

1) Methyl iodide and sodium carbonate:

The reaction was studied with a range of solvents, temperatures, reaction times and concentrations of methyl iodide. The optimum conditions involved heating methyl iodide (20 microlitres) and anhydrous sodium carbonate (5 mg) together with the parent acids and phenols in acetone (1 ml) in a capped vial at 55°C for 1 hour. Separation of the liquid phase, evaporation of the bulk of the solvent, followed by dilution with hexane furnished the derivatized analytes ready for injection onto the GC. Reproducible results, comparable to those obtained using diazomethane, were obtained over a wide range of analyte concentrations.

2) On-column Derivatization:

Chlorophenols and phenoxyacids were co-injected with Methelute (trimethylanilinium hydroxide) onto the GC columns

using a variety of Methelute concentrations, solvents and injection temperatures. This technique gave higher levels of interferences and generally the reactions were incomplete as compared to diazomethane. Picloram gave quite low yields of the methylated product.

3) Extractive Alkylation:

This technique was investigated using both tetrabutylammonium hydroxide (TBAH) and trimethylanilinium hydroxide as the counter-ion and with methyl iodide. The reaction was studied for a range of temperatures, reaction times and concentrations of methyl iodide and analytes. TBAH was found to be the most effective counter-ion. Although methylation by this method was more effective than on-column derivatization, it was less reproducible and gave generally lower yields than either diazomethane or methyl iodide/sodium carbonate, especially for picloram.

Conclusions:

1-The study on the use of SPE for the extraction of PAHs from water is continuing.

2-Replacement of diazomethane by methyl iodide/base in the derivatization step for the analysis of chlorophenols and phenoxyacids will be evaluated in field samples.

3-Attempts will be made to combine extraction with the derivatization step in the analysis of chlorophenols and phenoxy acids.

References:

1. MOE Method HPLC/L-X3HIDI. The determination of polynuclear aromatic hydrocarbons in surface water, domestic water and industrial wastes by HPLC.
2. MOE Method PWACP/PA. The determination of chlorophenols & phenoxy acids in effluents and water by GC-ECD.
3. Report No 2. MOE contract A94836. March 26, 1987.